




Optimization of ceramic-based noble metal-free catalysts for CO oxidation reactions

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Abstract

In this study ceramic supported noble metal-free catalysts promoting the oxidation of CO were examined. In the course of our work several non-noble metal containing catalysts were prepared with different metal content by the well-known wet impregnation method and their catalytic activities were analyzed by gas chromatography (GC) experiments in CO oxidation reaction. In addition to GC measurements, X-ray diffraction, scanning electron microscopy, BET and X-ray photoelectron spectroscopy tests were also performed on our samples. During our work we found that cobalt-loaded silica-alumina-based ceramic supported catalyst proved to be the best in CO oxidation due to the high activity and durability with comparable activity with Pt-loaded counterpart.

Keywords Catalyst · Carbon monoxide · Oxidation · Ceramic · Cobalt · Flue gas

Introduction

According to WHO reports nearly 7 million people die every single year from exposure to polluted air containing fine particulate matters (PMs) derive partly from flue gases [1] and more than 35 thousand people die from carbon monoxide poisoning each year worldwide [2]. Main sources of atmospheric pollutants are flue gases derived from transport and chimneys of factories and households, but harmful components could be converted using different types of catalysts. Noble metals (Pt, Rh, Pd) are typically used as catalysts for the treatment of flue gases, but they are very

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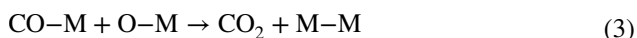
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expensive and available only in limited quantities, so many researches nowadays are therefore aimed at their substitutability.

Several institutes are focusing on the investigation of long-term trends to determine epidemiologic characteristics of the carbon monoxide-related deaths [3] and the reason of the high death risk [2] as well as greenhouse effect of such molecules [4, 5]. Oxidation of CO could be the process can be accelerated and the temperature conditions can be much more favorable by the help of adequate catalysts. Depending on the quality of the catalyst used the reaction can take place even at room temperature [6, 7]. During catalysis according to the Langmuir–Hinshelwood mechanism, the molecules of CO and O₂ are adsorbed on the surface of the catalyst, bound to the free valences of the metals and then on the surface CO and O₂ react to form CO₂, which is desorbed from the surface. This reaction process is illustrated by a series of reaction Eqs. 1–3 [8].



Michalak et al. found that addition of Sn to Pt provides a more efficient mechanism for CO oxidation compared to pure Pt due to the much lower activation barrier of PtSn catalyst derived from redox reaction (Sn²⁺–Sn⁰) that occurred at the interface of Pt and Sn oxide domains [9].

Experiment established by Moreau and Bond it was found that iron is an excellent promoter of gold catalysts and with the addition of 4 wt% iron content, they were able to improve the lifetime of the Au catalysts and the degree of deactivation process on several different supports (TiO₂, SnO₂, CeO₂, ZrO₂) compared to their Fe-free samples [10].

Dey et al. found that activity of the resulting catalyst is strongly dependent upon the combination of precursors because of the changing in the lattice oxygen mobility and calcination strategy which have great influences in CO oxidation activity [11].

Nowadays CO oxidation mainly achieved by transition metals instead of noble metals, because of the lower prices and availabilities, their efficiency is great, but influenced by several factors.

Soliman explained some of the factors affect the efficiency of catalysts in CO oxidation, based on the catalyst weight, particle size, pretreatment and preparation techniques, calcination temperature, and catalytic reaction temperature, which can also modify effects on the reaction [12]. Kim et al. developed a method for applying iron nanoparticles to a mesoporous alumina support. In addition, lifetime and temperature dependence of the Fe₂O₃/Al₂O₃ catalysts were also measured on several temperatures [13]. Selectivity is also important in the characterization of catalysts. Some studies examined metal oxide promoted noble metal catalyst for reducing CO in fuel processing applications without oxidizing any of the H₂ present [14, 15]. Numerous studies have already been performed to determine the role of water in oxidation reactions. Saveedra et al. also examined it in Au / TiO₂ systems and they

found that the presence of adsorbed water can enhance the catalytic activity where H_2O acts as a co-catalyst [16].

In a work published by Kwangjin An et al. the catalytic effect of mesoporous oxides and platinum nanoparticles were tested. They found that the combination of these two components acted as a more efficient catalyst because of the metal–oxide interfaces, where the most effective combination was $\text{Pt}/\text{Co}_3\text{O}_4$ [17].

Ferstl et al. identified possible adsorption sites and reaction mechanisms in Co_3O_4 -catalyzed CO oxidation which is aided by structural oxygen atoms of the Co_3O_4 , so that if too much oxygen leaves the Co_3O_4 , it recrystallizes to CoO [18].

Due to their huge specific surface, the number of active sites for adsorption is much higher during the catalytic use of nanoparticles, because accessibility is also easier for the components to be adsorbed. Kamel Eid et al. developed a method to form one-dimensional carbon-nitride nanofibers with Pt and Cu catalysts attached to it. Their studies confirmed that the Pt/Cu/CN NWs effectively catalyzed the conversion of carbon-monoxide to carbon-dioxide and were also able to preserve its durability and original structure [19].

It can be seen that the possible researches can be very extensive, many possibilities in the application and development of catalysts have not yet been mapped. Although researches that began nearly 200 years ago has answered many questions, it is still worth experimenting in this area, mainly on the field of non-noble metal and nanoparticle catalysts.

Experimental methods and materials

Materials

Cobalt(II) nitrate hexahydrate ($\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\geq 98\%$); nickel(II) nitrate hexahydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\geq 98.5\%$); gallium(III) nitrate hydrate ($\text{Ga}(\text{NO}_3)_3 \cdot \text{H}_2\text{O}$, $\geq 99\%$); copper(II) nitrate hydrate ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$, $\geq 99\%$); zinc(II) nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\geq 99\%$); iron(III) nitrate nonahydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, $\geq 98\%$) were manufactured by Sigma Aldrich.

Alumina (Al_2O_3 , $\geq 95\%$) was manufactured by MAL Ltd. Hungary; talc ($\text{Mg}_3(\text{Si}_4\text{O}_{10}(\text{OH})_2)$, $\geq 85\%$) and silica (SiO_2 , $\geq 99\%$) was traded by Imerys Refractory Manufacturer Ltd. Hungary and clay ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 + 2\text{H}_2\text{O}$, clay minerals $\geq 85\%$) was manufactured by Keraclay Plc. Czech Republic.

Ceramic support preparation

Using the appropriate quantities of raw materials (alumina, talc, silica and clay) a dense clay mass was prepared by adding water to it, which was homogenized by repeated kneading and the clay components were allowed to unfold for a week.

The clay mass was then fired at elevated temperature ($> 1000\text{ }^\circ\text{C}$) then the resulting ceramic was ground to fine powder.

Catalyst sample preparation

Our catalyst samples were prepared by the so-called wet impregnation method. The purpose of this method is to create an interaction between the metal precursor and the ceramic support, in which the impregnating solution is added in excess with the pore volume of the ceramic support with continuous stirring. During the preparation of our samples the nitrate salts of our metal precursors were dissolved in distilled water using an ultrasonic sonication bath in such an amount that the resulting samples were 0.1–0.2–0.5 and 5.0 wt% of metal ions for 500 mg of pre-annealed ceramic support. After that we added the ceramic powder to the solvent with continuous stirring and the distilled water was evaporated from the solution using a magnetic stirrer with continuous stirring at 350 rpm and heated to 90 °C. After the evaporation of water from the mixture our samples were placed into an oven for 12 h preheated to 80 °C in each case.

Characterization methods

Gas chromatography

The activity of our catalyst samples was determined by gas chromatography (GC) on a Hewlett Packard 5890 Series II gas chromatograph. During the measurements, 250 mg powder samples were placed in the middle region of a 1 mm wall thickness, 8 mm internal diameter, 250 mm long quartz tube, which was inserted vertically into a tube furnace. Before starting the measurement, the samples were pretreated by oxidation and then reduction. During the oxidation, the sample was purged with pure O₂ gas at 300 °C for 30 min, thus the metal nitrate salts in the ceramic powder support were then oxidized, leaving the system in the form of nitrous gases while the metals remain on the surface of the ceramic support in oxidized state. After oxidation, the system was purged with pure Ar gas for 10 min. After the oxidative pretreatment and argon purging the samples were pretreated by reduction. During the reduction, the sample was purged with pure H₂ gas at 300 °C for 60 min, for removing unwanted surface poisons as well as forming the active Co-oxide phases. The entire system was then purged again with Ar gas for 10 min.

After the pretreatment processes of the catalyst samples our system was cooled back to 373 K and then the measurements were performed using a CO–O₂–Ar reaction gas mixture at 7 measuring points from 373 to 973 K. The flow rate of our reaction gas mixture was 4–10–46 mL/min during the measurement respectively. The detector signal was provided by the thermal conductivity detector (TCD) based on the different thermal conductivity of the components.

X-ray diffraction (XRD)

A Rigaku MiniFlex II X-ray diffractometer was used to analyze the composition of our samples. The samples were analyzed at a measurement speed of $2^\circ/\text{min}$ for the highest possible resolution. During the measurements Cu K_α anode X-ray source was used.

Scanning electron microscopy (SEM) with energy dispersive X-ray spectrometer (EDS)

The instrument used for the investigation was a Hitachi S-4700 scanning electron microscope (SEM) with a cold field emission (CFE) cathode. The energy dispersive X-ray spectrometer (EDS) attachment was a Röntec QX2 spectrometer. The system was used under 10^{-7} Pa ultra-high vacuum and the samples were maintained at room temperature and under 10^{-4} Pa vacuum during characterization in the sample chamber. The accelerating voltage was 15 kV.

X-ray photoelectron spectroscopy

Measurements were performed using a KRATOS XSAM 800 instrument, which was used under ultra-high vacuum conditions with an Mg K_α anode X-ray source operated at 144 W.

Results and discussion

To select the appropriate catalyst synthesis method we examined the activity of two catalyst made by different techniques and we also tested the activity of the pure ceramic support marked by sample (a) as a standard. The first catalyst we tested was sample (b) containing 0.5 wt% Co(II) where cobalt ions were supported onto the ceramic surface before the high temperature heat-treatment of the ceramics. The second one was sample (c) containing 0.5 wt% Co(II) where cobalt precursor was applied onto the ceramics after the same high temperature heat-treatment required for the ceramic support. Gas chromatographic measurements were performed as previously described, the results are shown in Fig. 1, where the temperature and time dependence of CO conversion can be seen.

Based on Fig. 1A, the efficiency of catalytic activity can be clearly distinguishable for the three samples. While the CO conversion at 473 K was 92% in the case of pre-annealed ceramic support containing sample (c), both post-fired ceramic support containing sample (b) and pure heat treated ceramic support sample (a) had no catalytic activity on the same temperature. The efficiency of the pre-annealed sample was persistent as a function of increasing temperature above 473 K. The activity of pure ceramic increases almost linearly with the temperature and at the highest temperature its conversion rate achieved the maximum efficiency of 40–45%. In

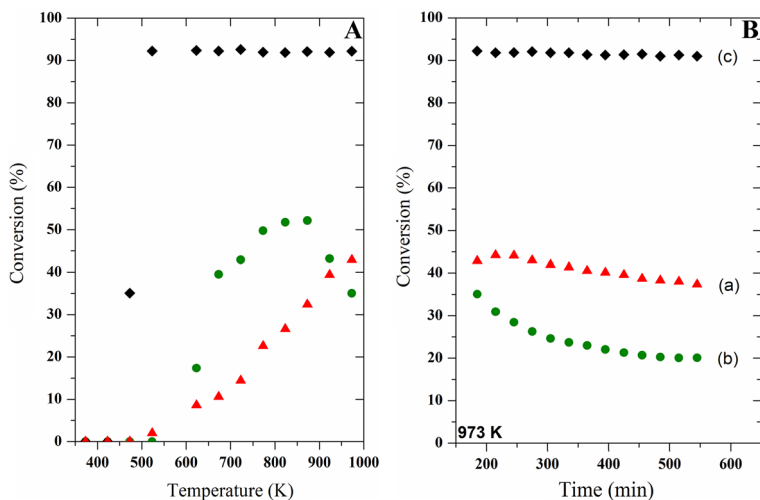


Fig. 1 CO conversion as a function of temperature (**A**) and time on elevated temperature (**B**) for different catalyst support samples including pure ceramic where (a) is the pure and heat-treated ceramic support, (b) is the raw ceramic support with 0.5 wt% Co(II) without heat-treatment and (c) is the heat-treated ceramic support with 0.5 wt% Co(II)

contrast, the activity of the post-annealed sample was also linear with the temperature until a conversion rate of 52% but suddenly started to decrease above 873 K until it reached a conversion rate of 35% as a function of increasing temperature which means less conversion compared to the pure ceramic support.

Investigated the temperature dependent catalytic activity of pure ceramic support it can be concluded that it was not only served as a support surface for metal ions but also participated in the process of CO oxidation. From Fig. 1A it can be also clear that from the two synthesis methods, pre-annealing of the ceramic support is the proper process. During post-annealing process, the metal ions were also exposed to high-temperature treatment on the surface of the ceramics, which appears to have a disadvantageous effect during subsequent experiment. Presumably, as a result of high temperature treatment, the structure and crystal size of the metal catalyst changed and aggregated on the surface of the ceramic support, which significantly reduces the activity of the catalyst. In the case of pre-annealing, metal ions were not exposed to such a high temperature, only the ceramic support, so the catalyst could keep its specific surface, thus providing active sites for catalysis.

Fig. 1B shows the results of CO conversion in the function of time at elevated temperature at 973 K for 9 h where the aim of the measurement was to determine the effect of persistent high temperature on the activity of the catalysts. In the case of sample (a) and sample (b) a slight decrease can be seen in the conversion and only sample (c) was permanent as a function of time at high temperature, so we used this synthesis method in the future where catalyst precursors applied onto the surface of the ceramic support only after its heat-treatment.

X-ray diffractogram obtained on the heat-treated ceramic support is illustrated in Fig. 2. The details of the measurement between 15 and 80 2θ degrees can be seen

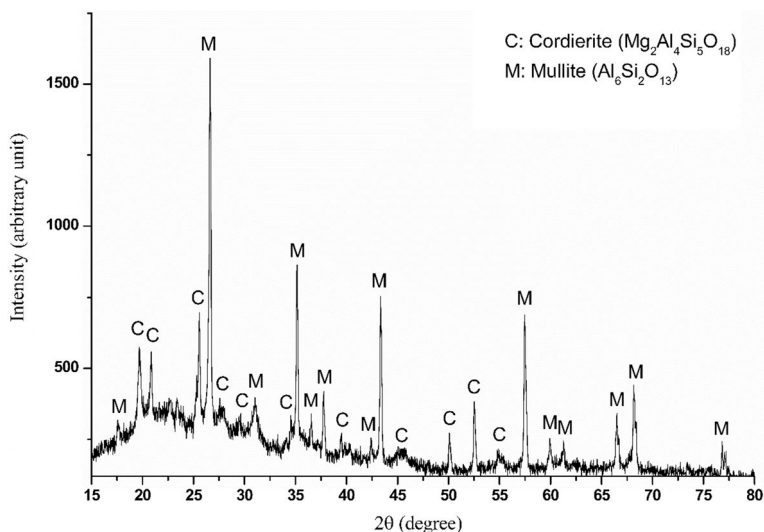


Fig. 2 XRD patterns of ceramic support powder where C is α -cordierite and M is mullite

in the figure where cordierite reflections were marked with C and mullite reflections with M. The phase composition of ceramic support was presented in Fig. 2, which showed mainly α -cordierite ($\text{Mg}_2\text{Al}_4\text{Si}_5\text{O}_{18}$) and mullite ($\text{Al}_6\text{Si}_2\text{O}_{13}$) phases in the ceramic support.

The structure and size of the different particles in our ceramic support was determined by scanning electron microscopy. Using a magnification of $\times 1000$ or $\times 5000$ grains became clearly visible on Fig. 3.

Dimensions of the grains were measured with ImageJ analyzer software and afterward we determined the average particle size of the ceramic support as shown on Fig. 4.

We extended our measurements to other non-noble metal catalysts to see which one could be the best suited for CO oxidation under the same conditions.

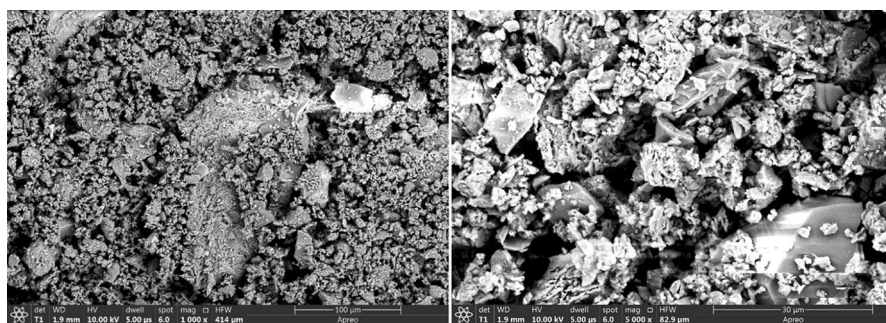


Fig. 3 SEM images of the ceramic support using a magnification of $\times 1000$ and a scalebar of $100\ \mu\text{m}$ (left) and a magnification of $\times 5000$ and a scalebar of $30\ \mu\text{m}$ (right)

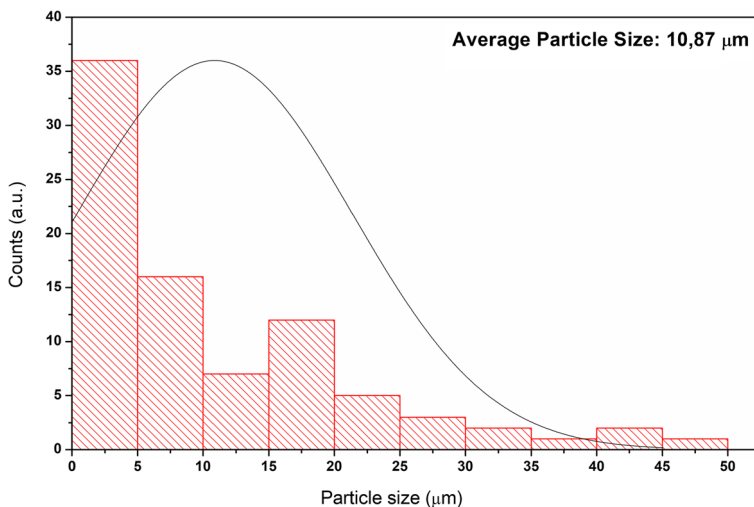


Fig. 4 Histogram to define size distribution and average particle size of the ceramic support

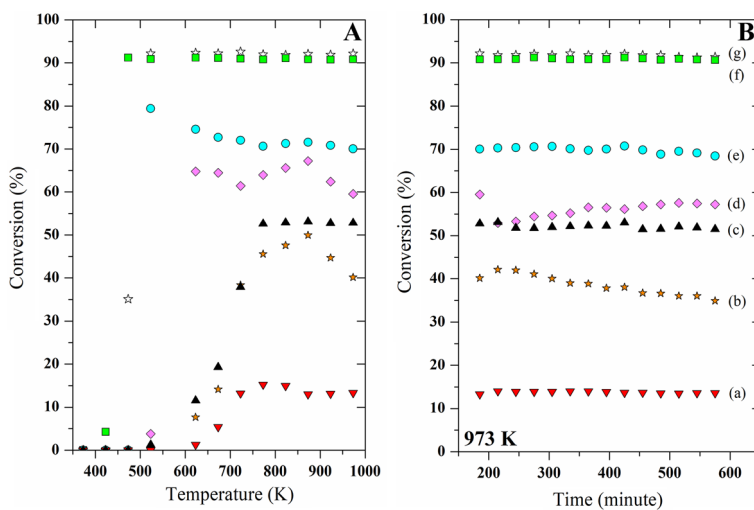


Fig. 5 CO conversion as a function of temperature (A) and time at elevated temperature (B) for different catalyst samples with an amount of 0.5 wt% metal ion on the ceramic support where (a) is Zn(II) nitrate, (b) is Ga(III) nitrate, (c) is Fe(III) nitrate, (d) is Ni(II) nitrate, (e) is Cu(II) nitrate, (f) is Pt-salt as reference and (g) is Co(II) nitrate

Thus, in addition to the previously tested cobalt(II) nitrate precursor, we examined nickel(II) nitrate, gallium(III) nitrate, copper(II) nitrate, zinc(II) nitrate, and iron(III) nitrate precursors. The catalyst samples were prepared in each case with a metal ion content of 0.5 wt% the results of the measurements are shown in Fig. 5.

It can be seen in Fig. 5A that the best results were obtained with sample (g) catalyst prepared from Co(II) nitrate. The second one was sample (e) catalyst made of Cu(II) nitrate precursor. The efficiency in CO conversion of sample (e) was 20% lower than that of the Co(II) containing one, so the copper-containing catalyst can also provide us a suitable basis for future researches.

Fig. 5B shows the durability of the samples at 973 K for 10 h, from which it can be concluded that all ceramic-supported metal catalysts proved to be stable. The basic task of our ceramic support is to ensure the maintenance of the dispersion of metal particles, but it also serves the purpose of protection against poisoning and sintering caused by higher temperatures. Since the service life of each catalyst sample proved to be durable during the measurements, it can be concluded that the ceramic used as a support is suitable for this kind of task.

Overall, based on our results we can state that sample (g) containing 0.5 wt% Co(II) was the most efficient catalyst so we subjected it to further investigations with different amounts of Co(II). The results of these experiments are shown in Fig. 4, where the concentrations of the examined samples was 0.0–0.1–0.2–0.5–5.0 wt% Co(II) on the ceramic support.

Fig. 6A shows the differences in the catalytic activity among the samples containing Co(II) in different amounts. Sample (e) containing 5.0 wt% Co(II) had the highest catalytic activity with a conversion of 93–95% which was achieved at the temperature of 473 K and this activity was stationary as a function of increasing temperature and reaction time. Just like the following sample (d) containing 0.5 wt% Co(II), which was capable of nearly 90–92% conversion with increasing temperature and its activity was also stable as a function of reaction time.

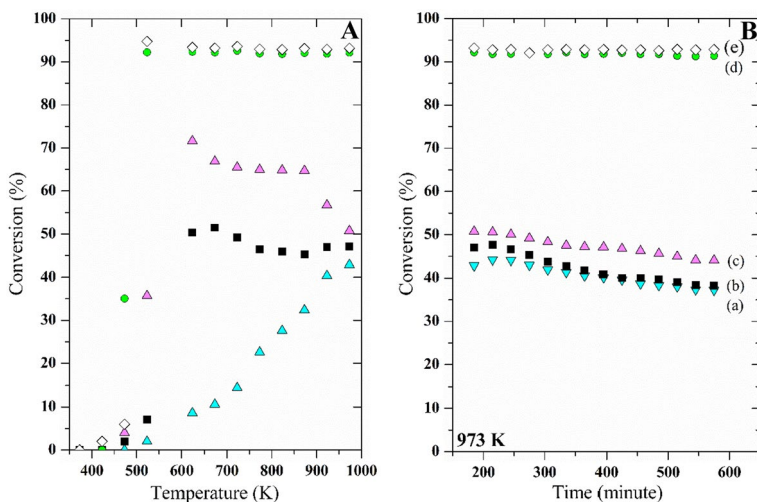


Fig. 6 CO conversion as a function of temperature (**A**) and time on elevated temperature (**B**) for different amount of cobalt catalysts samples on ceramic support where (a) is the pure ceramic support without any catalyst, (b) is the sample containing 0.1 wt% Co(II), (c) is the 0.2 wt% Co(II), (d) is the 0.5 wt% Co(II) and (e) is the 5.0 wt% Co(II) ions

In the case of sample (b) containing 0.1 wt% Co(II) and sample (c) containing 0.2 wt% Co(II) the rate of CO conversion significantly increased in the function of increasing temperature at lower temperatures but broke at 623 K and started to decrease to 50% in the function of rising temperature. In the function of time the conversion ratios of these two samples continued to decline to 40–45%. After circa 4–5 h the catalytic activities of these two samples was equal to the pure annealed ceramic support until the highest examined temperature which was also slightly declined in the function of reaction time up to 37%.

Further analyzing Fig. 6A, it can be seen that that the higher the Co(II) amount on the surface of the ceramic support, the higher the conversion rate we could achieve.

However, in the case of sample (d) containing 0.5 wt% Co(II), a significantly higher activity can be achieved, which can be assumed due to the fact that the high concentration of the precursor results in a larger particle size on the ceramic support surface, so the resulting specific surface area of the catalyst will be smaller and the activity decreased. Although the observed catalytic activity of sample (e) containing 5.0 wt% Co(II) was higher than the activity of sample (d) containing 0.5 wt% Co(II), there was a tenfold difference between the amounts of Co(II) applied to the ceramic substrates. Therefore, taking into account the economic and financial aspects, it is more worthwhile to decide on the development of the 0.5 wt% Co(II) containing catalyst, as its production cost is much lower, but its efficiency quite similar to its counterpart with 5.0 wt% amount of Co(II). Fig. 6B shows the results of the time dependent part of our measurements at the temperature of constant 973 K. It can be stated that no significant decrease can be seen in the conversion and each samples retained their applicability and remained persistent even at elevated temperatures.

For better understanding of the high activity of the cobalt containing samples, the composition of the pure ceramic was summarized in Table 1, while the composition of the sample containing Co(II) is shown in the same table. The pure ceramic sample contained 55.45 wt% of silicon and 35.13 wt% of aluminum. This was consistent with the XRD measurement performed previously, where the SiO₂ and Al₂O₃ containing components of the support were detected by X-ray diffraction analysis. In addition, the samples contain magnesium (4.46 wt%), potassium (1.54 wt%), iron (1.46 wt%), titanium (1.2 wt%) and calcium (0.75 wt%)

Table 1 Results of SEM–EDS analysis of ceramic support and 0.5 wt% Co(II) containing catalyst

	Pure ceramic support	0.5 wt% Co(II) containing sample
	Norm. wt%	Norm. wt%
Magnesium	4.46	4.20
Aluminium	35.13	34.77
Silicon	55.45	52.74
Potassium	1.54	1.64
Calcium	0.75	0.79
Titanium	1.20	1.43
Iron	1.46	1.48
Cobalt		2.95

as contaminants. Previous measurements have shown that the pure, catalyst-free ceramic support was also catalytically active, which may be explained by the presence of these contaminants as promoters. The catalytic activity of Fe and Ti in CO oxidation reactions has been investigated in several studies and the results have shown their catalytic activities on their own and when used as promoters.

The second column of the table shows the SEM–EDS results of the catalyst containing 0.5 wt% Co(II). Obviously, this sample is also containing the same elements in almost the same amounts as in the pure ceramic one, but in this case the cobalt was also detected in the sample.

The specific surface area of these samples were determined based on the BET theory, and the surface area of the ceramic support was 4.1 m²/g and that of the cobalt-containing catalyst is 3.6 m²/g. The size of the active surface was smaller compared to data in the literature, where for example in the case of Pt, Pd and Rh catalysts the specific surface area can be 80 m²/g, in the case of Co₃O₄ catalysts 43–143 m²/g, and in the case of nanocatalysts hundreds of m²/g size [8]. Even so, our measurements confirmed that a high stability and poison-resistant catalyst was successfully synthesized with the basics of the special mullite/cordierite supported catalysts.

Finally, the 0.5 wt% Co(II) containing catalyst was also analyzed by X-ray photoelectron spectroscopy (XPS) to define the oxidation states of the elements in the sample as we can see in Fig. 7.

Based on the results above, the following peaks were identified in ascending order: 74.5 eV is aluminosilicate 2p peak, 102.2 eV is silicone oxide 2p peak, 102.8 eV is aluminosilicate 2p peak, 284.8 eV is carbon contamination 1 s peak, 529.8 eV is aluminosilicate/residue cobalt(II) nitrate peak, 780.1 eV is Co(II) oxide 2p peak and 795.9 eV is also a Co(II) oxide 2p peak.

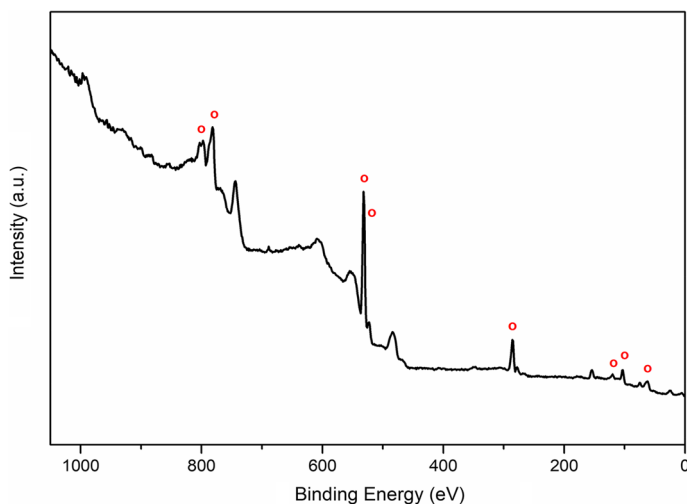


Fig. 7 X-ray photoelectron spectroscopy of the 0.5 wt% Co(II) containing catalyst

The presence of Co(II) 2p peaks according to the strength of the binding energy detected from the sample suggests that the cobalt(II) nitrate initially supported onto the surface of support was converted to cobalt(II) oxide by the heat treatment. In terms of catalytic activity, the presence of cobalt(II) oxide is authoritative, as many studies have shown that the presence of oxygen in the catalyst supports the process of CO conversion.

Summary

The aim of our study was to determine the optimum concentration, where a non-noble metal should be supported onto the surface of a ceramic support to create a catalyst for the efficient oxidation of carbon monoxide.

We investigated the catalytic activities of the non-noble metal based ceramic supported catalysts and after the comparison of the different the samples we found that the Co(II) containing ones had the best efficiency. Moreover, the activity of the best cobalt containing sample was comparable with the activity of the Pt-loaded counterpart.

The durability of the catalysts were also examined by temperature- and time-dependent gas chromatographic studies where catalysts resisted the permanent high temperature and retained their catalytic activity.

Based on the facts above, it can be stated that the 0.5 wt% Co(II) containing sample should be used and further developed in CO oxidation processes from both economic and environmental point of view.

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